PROJECT NUMBER : 6505
PROJECT TITLE : Special Investigations
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I. MENTHOL

A. Objective: To develop methods for the quantitation of menthol release compounds such as beta-1-menthyl itaconate, monomer and polymer, and POLYMIC. To utilize these methods for sample analysis as required.

B. Results:

A capillary GC procedure was developed for POLYMIC using the response of menthol for quantitation. Menthol was released from POLYMIC by refluxing with an ethanolic/KOH solution. POLYMIC was determined in two filler samples (J1AFU and J1AFV) having a target application rate of 2.7%. The levels of POLYMIC measured in the samples using this method were 2.4% and 2.7% by weight, respectively.

Menthol released from a POLYMIC hot-melt sample was determined by comparison to a POLYMIC "powder" standard. Menthol was released from both samples as described above. The menthol response of the POLMIC hot-melt was 66% of the menthol response for the POLYMIC standard. These results included any free menthol which may have been present in the hot-melt prior to refluxing.

Poly(β-1-menthyl itaconate) (POLYMIT) was determined in loose filler (J1AKJ) and corresponding cigarette filler (X6D1BWL) having a target application rate of 4%. The POLYMIT levels measured in these samples were 4.1% and 4.3% by weight, respectively.

C. Plans: Plans are to analyze samples for such menthol release compounds as required. Methods will be developed as needed including the evaluation of purge and trap techniques.

II. R&D AND OPERATIONS SUPPORT

A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

An HPLC method was developed for the determination of glycyrrhizic acid in tobacco in support of Marlboro Standardization. The method combined the sample preparation procedure from R&D Method E-78 (by G. Carter) and the

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chromatographic conditions of Flavor Specifications Method #6 (by M. Zimmermann). Approximately 100 tobacco samples were subsequently analyzed using the modified procedure.

A "mini" training course was provided for five representatives from four tobacco suppliers on HPLC basics and the HPLC determination of methoprene. The suppliers are in the process of setting up the methoprene method in their laboratories for routine testing of tobacco purchased for Philip Morris.

A study to evaluate chemical changes leading to "stale" cigarettes continues. Conditions were established to reproducibly reduce the water content of Marlboro cigarettes from 11.5% to ~9% using vacuum drying, desiccant ($CaSO_4$), nitrogen sweep, and an oven. Re-conditioning of the cigarettes to bring their water content back to 11.5% is in progress in preparation for subjective evaluation scheduled for July 29.

Jaguar CB10 was examined using Curie Point Pyrolysis followed by GC/MS of the volatile pyrolyzate. The material was pyrolyzed and 315°C and 590°C. The major compound observed at both temperatures was 1-hydroxy-2-propanone. Other compounds observed included ketones and hydroxy compounds. Jaguar CB10 is under evaluation as a potential binder for the new cast leaf process.

Nicotine was determined in XAD tubes through which CO_2 had been vented from the NET process and also in methanol washes of the corresponding CO_2 cylinders. Nicotine was also determined in isopropanol washes of vented CO_2 cylinders from the current DIET process. Data were reported to E. Thomas.

In support of an ethanol accountability study in progress at Cabarrus, a series of traps were evaluated for efficiency and stability of ethanol containment. Ethanol vapors were drawn through two methanol, one silica gel, and two charcoal traps (in series) using a vacuum induced air flow (2 L/min) over the ethanol. Results showed that 97% of the ethanol was trapped in the two methanol traps. A recommendation for trapping ethanol vapors was made accordingly.

Three DBC Bright samples were analyzed for propionic and butyric acids by capillary gas chromatography. None of the samples contained either of the acids which are typically used to monitor spoilage. The limit of detection was approximately 10 μg/g for both acids under the operating conditions specified. This preliminary evaluation was in support of the mold issue.

Fifteen RCB samples were analyzed for butyric acid by capillary gas chromatography. Butyric acid was not detected in any of the samples except one which contained < 50 μg/g. The limit of detection was approximately 10 μg/g. Spoilage was being monitored in these samples as part of the ongoing RCB Stability Program.

Eighteen CEL and SEL samples were analyzed for propionic acid to monitor potential spoilage in the new Weigand Evaporator. Determination was made by capillary gas chromatography. Levels of propionic acid in the SEL samples ranged

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from none detected to about 25 μg/g. In the CEL samples, levels ranged from about 60 to 100 μg/g. The limit of detection of propionic acid in the SEL was approximately 10 μg/g under the operating conditions specified.

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